

**ELECTRICAL CHARACTERIZATION OF GaAs SINGLE CRYSTAL  
IN DIRECT SUPPORT OF M555 FLIGHT EXPERIMENT**

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CHARACTERIZATION OF GaAs SINGLE CRYSTAL IN  
DIRECT SUPPORT OF M555 FLIGHT EXPERIMENT  
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by

**J. G. Castle**

**Final Technical Report**

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September 1975



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**FINAL TECHNICAL REPORT ON CONTRACT NAS8-29542**

**TITLE OF**

**CONTRACT:**

**Electrical Characterization of GaAs Single Crystal in  
Direct Support of M555 Flight Experiment**

**SUBJECT:**

**Electrical Characterization of Single Crystal Surfaces**

**PREPARED BY:**

**J. G. Castle, Principal Investigator  
Professor of Physics, UAH**

**ABSTRACT:**

The exploration of several nondestructive methods of electrical characterization of semiconductor single crystals was carried out during the period ending May 1974. Two methods of obtaining the microwave skin depth, one for the mapping flat surfaces and the other for analyzing the whole surface of small single crystal wafers, were developed to the stage of working laboratory procedures. The preliminary 35 GHz data characterizing the two types of space-related single crystal surfaces, flat slices of gallium arsenide and small wafers of germanium selenide, are discussed. A third method of nondestructive mapping of donor impurity density in semiconductor surfaces by scanning with a light beam was developed for GaAs; its testing, done to a large extent without NASA support, indicates reasonable precision at reasonable scan rates for GaAs surfaces at room temperature.

**SUBMITTED TO:**

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School of Graduate Studies and Research  
P. O. Box 1247  
Huntsville, AL 35807**

**September 1975**

FOREWORD

The author gratefully acknowledges the many significant contributions of his supporting staff on this remarkably successful project. Gratitude goes: to R. R. Lattanzi, Research Engineer and Paul Zimmerman, Electronic Technician for their careful learning of some microwave techniques,

to James M. Rowe, Graduate Student, for his diligence in the successful optical scanning of GaAs at room temperature

to Michael Valpiani, Senior, for his effective derivation of the algebra and execution of data reduction for the 35 GHz skin effects for this final report and

to Karla Dalton, Sophomore, for her mastery of the programming of the new graphics display terminal so as to display the luminescence spectra automatically,

but any shortcomings in this report rest with the P.I. The reader is warned that NASA's kindness in allowing a one-year, no-cost extension for the purpose of improving the write-up does not guarantee the correctness of the formulae presented herein. The 35 GHz skin depth values were calculated from observed power ratios by the use of these formulae for data reduction and were submitted to the COR early in the summer of '74.

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## I. INTRODUCTION

The NASA program for growth of semiconductor single crystals in the space environment of Skylab was expected\* to produce crystals of significantly higher quality than attainable through the same growing processes on earth. As the semiconductor crystals become more perfect if undoped or more uniform if doped, the emphasis in any measurement of them shifts increasingly toward care in avoiding damage to the crystal by the measurement.

### A. Need for Nondestructive Method for Electrical Characterization

Electrical characterization of a single crystal of semiconductor usually involves "soldering" ohmic contacts in several locations in order to obtain some average bulk property (e.g. bulk resistivity) for the sample piece. Whenever the crystal is to be evaluated for use in large scale integrated (LSI) circuitry, the characterization should include a search for local variations in electrical properties. Such a search should use a nondestructive method of mapping the surface of the semiconductor.

Clearly, the mapping of electrical characteristics of high quality crystals, such as those grown on Skylab flights, should be done without inflicting any damage. To this end, noncontacting methods of mapping were sought - avoiding the formation of "solder" contacts and avoiding the high local pressure of point contacts. In addition to valuable monitoring of NASA's space processed materials, the successful development of suitable noncontacting methods of electrical measurement would be of considerable spinoff value to U. S. semiconductor manufacturers or at least to U. S. semiconductor circuit manufacturers.

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\*This expectation was realized many times in high quality semiconductor crystals grown on the Skylab missions during 1973 and 1974.

### B. Noncontacting Techniques for High Quality Single Crystals

The class of techniques by which the electrical properties of a semiconductor single crystal can be observed without contacts is limited to coupling to the crystal by electromagnetic radiation at frequencies from rf to the near uv. The optical frequencies have been used to probe the uniformity of certain types of impurities in semiconductor crystals.<sup>1</sup> A wide range of radiation frequencies can be used to observe the mobilities and the concentrations of the various types of charge carriers present near the surface of the semiconductor crystal being examined. For surveying carrier concentrations and mobilities of thin semiconductor sheets, useful measurements of optical transmission and reflection with commercial instruments have been reported<sup>2</sup> with 1.5 mm diameter resolution. In addition, the impurities within the semiconductor can often<sup>3</sup> be identified by X-radiation.

Coupling to a semiconductor at high frequencies implies<sup>4</sup> the reaction of the semiconductor will take place near the surface, i.e., within the "skin depth,"  $\delta_s$ . The "classical" application of Maxwell's equations for the case of a field  $E_z$  applied to a plane surface of semi-infinite conductor extending from  $x = 0$  gives the current density varying with the depth  $x$  as<sup>5</sup>

$$J_z = \sigma E_z e^{-x/\delta_s} e^{-ix/\delta_s} \text{ amp/m}^2, \quad (1)$$

where the normal skin depth,  $\delta_s$ , is defined as

$$\delta_s = (\rho/\pi f \mu_0)^{1/2} = 504 (\rho/f)^{1/2} \text{ meters} \quad (2)$$

for  $\rho$  in ohm-meters and  $f$  in Hz. The resistivity, for a semiconductor whose dominant charge carrier has an effective mass  $m^*$  and mean-free time between collisions of  $T$ , can be expressed<sup>6</sup> by the product of  $T$  and the carrier concentration  $n$  as

$$\rho = \frac{m^*}{e^2} \frac{1}{nT} . \quad (3)$$

Usually  $T$  and  $n$  are each significant in characterizing the crystal.

It might be noted in passing that this normal skin depth is small in our best metallic conductors even at power line frequencies. For example, Equation 2 indicates, as is found in practice, that  $\delta \approx 7$  mm in copper and in aluminum at 60 Hz and at room temperature.

#### 1. Use of Radio Frequencies to Microwaves

Returning to the choice of method to characterize a flat semiconductor crystal by its resistivity in the volume encompassed by the depth of  $x \approx \delta$  below the surface, we can choose the frequency to use in order to probe the predetermined depth,  $\delta$ . Likewise, we can choose the frequency to obtain a reasonable resolution of the mapping since the minimum surface area (= resolution element) that can be probed measures approximately one-half wavelength across. In any case, an instrumental measurement of a value for the skin depth at a given frequency will give, by Equation 2, the effective resistivity near the crystal surface, averaged over the area exposed to the radiation.

For conducting crystals, the normal skin effect applies to within a few percent<sup>7</sup> with the mean-free path of the charge carriers equal to one skin depth as given by Equation 2. Values for the normal skin depth are, by Eq. 2:



For resistivity of 10 ohm-cm,  $\delta_s = 16$  mm at 100 MHz,  
 $\delta_s = 1.6$  mm at 10 GHz, and  
 $\delta_s = 0.8$  mm at 35 GHz.

For resistivity of 0.001 ohm-cm,  $\delta_s = 0.16$  mm at 100 MHz, and  
 $\delta_s = 0.008$  mm at 35 GHz.

Values to 60 GHz are given for silicon in a recent book by H. F. Matare.<sup>8</sup> Comparison to carrier mean-free path,  $\ell$ , can be made by the "free electron" model<sup>6</sup> for our semiconductor crystal by estimating the Fermi velocity,  $v_F$ , to be  $1(10^7)$  cm/sec for  $m^* = m_e$ . The expression for mean-free path is

$$\ell = v_F T.$$

Values of  $T = m^* \mu_H / e$  depend on the Hall mobility,  $\mu_H$ . So the values of mean-free path,  $\ell$ , expected for "free" electrons in our semiconductor at room temperature are estimated to range from  $\ell = 0.4$  micron for  $\mu_H = 10^4$  cm<sup>2</sup>/Volt sec to  $\ell = 0.04$  micron for  $\mu_H = 10^3$  cm<sup>2</sup>/Volt sec. Clearly, we expect  $\ell \ll \delta_s$ .

Therefore, the classical expression for the skin depth ( $\delta_s$  by Eq. 2) should be an accurate way of calculating the resistivity of flat semiconductor samples from measurement of skin depth,  $\delta$ , even at frequencies higher than 35 GHz.

Cyclotron resonance and electron spin resonance can give useful characterization within the skin depth region too. For example, the concentrations of impurities in Ge were recently measured<sup>9</sup> by cyclotron resonance.

## 2. Use of Optical Radiation

Coupling to a semiconductor at optical frequencies can yield electrical characteristics.<sup>10</sup> For thin semiconductor wafers, standard optical (infrared) measurements of reflection and transmission have been used to map carrier concentration and mobility; the report by Black, et al.<sup>2</sup> indicates precision better than 10% over a wide range of values for GaAs wafers scanned with a 1.5 mm diameter beam on a Perkin Elmer Model 621 recording spectrophotometer.

NASA's needs include the ability to characterize the surface of more massive semiconductor crystals - possibly too thick to obtain infrared transmission values as above. For certain semiconductors, like Si and Ge, edge contacts will serve to obtain a map of photovoltaic (PV) response from a spot of light impinging on the crystal surface, revealing nonuniformity of carrier concentration and/or mobilities. Meaningful PV response has apparently not yet been attained on other semiconductor crystals, such as GaAs. For large band gap semiconductors like SiC and GaAs, the light emitted by electron-hole recombination processes has been used<sup>11,12</sup> to identify the electrically active impurity sites present. Our success at obtaining impurity concentration in volumes of GaAs less than a cubic millimeter is mentioned in the Results Section.

Another class of optical techniques which has promise for electrical characterization involves photo-induced changes. We looked briefly for photo-induced conductivity at room temperature,<sup>13</sup> but our search was inconclusive. Photo-induced microwave conductivity is still a potentially useful technique for mapping high quality semiconductor crystals without contacts.

### C. Crystal Selection for this Study

The crystals of semiconductors used in this study of nondestructive techniques for observing electrical characteristics were supplied through the good offices of the NASA Space Sciences Laboratories, MSFC. The emphasis during the earlier portion was on characterizing flat surfaces of GaAs in preparation for the M555 Flight Experiment. The emphasis during the last six months was on small single crystalline flakes of vapor-grown germanium selenide. The comparison of 35 GHz skin depths we observed in GeSe flakes grown<sup>14</sup> on Skylab to GeSe flakes grown at RPI is tabulated below.

### D. Purpose

It was, therefore, the general purpose of this contract to demonstrate the sensitivity of one or more noncontacting methods of electrical characterization of single crystals of the semiconductors to be grown on Skylab. Specific goals dealt at first with characterizing surfaces of gallium arsenide slabs and dealt in the last half year with small flakes of germanium selenide single crystals.

Single crystal samples to be studied were supplied through the Space Sciences Lab of NASA/MSFC. The specific approaches that we investigated included microwave skin depth and e-h recombination luminescence. Two microwave techniques were developed to the point of collecting statistical evidence of sensitivity and reproducibility. The GaAs luminescence scanning technique was developed by an interested graduate student J. M. Rowe, largely on his own time.

## II. APPROACH TAKEN

### A. Noncontacting Techniques Considered

#### 1. RF Resistance by Coil Loading

The eddy currents induced in a semiconductor specimen are a specific but complicated geometric function of shape of coils and semiconductor and of the uniformity of resistivity. An example of successful noncontacting measurements of bulk resistivity by eddy currents at 10 MHz was reported by J. C. Brice and P. Moore in J. Sci. Inst. 38 (1961) on page 307. Mapping could be accomplished with sufficiently small coils but consistent coupling is a difficult mechanical problem.

#### 2. Surface Effects at 9 GHz and 35 GHz

Preference was given to 35 GHz because the area of the mapping resolution element can be smaller by  $\times 16$ . We considered the surface resistance by waveguide termination<sup>15</sup> and by cavity loading, preferring the latter for flats with high resistivity and for the small flakes. We considered the microwave Hall effect. After several tries with biomodal cavities, we became more fully appreciative of the critical need for equivalence in the degenerate modes as called for by A. M. Portis in his Phys. Rev. paper, a truly severe mechanical challenge and, therefore, not suitable for rapid surveying of flat samples.

Cyclotron resonance remains a promising prospect<sup>9</sup> for measuring the lifetime of each type of carrier provided the mapping can be arranged at low temperatures.

Photo-induced changes in microwave conductivity (PC) similarly stands as a promising non-contacting way<sup>13</sup> of mapping several electrical characteristics with a resolution element approaching the size of the minimum light beam. Photo-induced microwave conductivity should be especially useful in semiconductor samples of very high resistivity for which the sensitivity of surface resistance measurements is declining.

### 3. Optical Means

Fluorescence from electron-hole recombination is frequently a direct indicator of the donor impurity density.<sup>10-12</sup> The literature is literally crowded with publications of such observations for semiconductors at low temperatures. We found little evidence of successful fluorescence at room temperature so we took up that challenge.

Raman scattering from defect modes at the impurities of interest is weak at room temperature being usually not sensitive enough to reveal the impurity concentrations of interest even at very low temperatures.

#### B. Noncontacting Techniques Used

Each technique for noncontacting electrical characterization of semiconductor surfaces was chosen to demonstrate its reliability for absolute values in a sample sequence representative of the short term needs of NASA's program for space crystallization. Repeated substitution of samples was employed to attain preliminary statistical evidence of reliability.

It should be noted that in any sequential substitution method there are sources of dc noise which can be reduced or eliminated by properly engineered modulation techniques. Therefore, the reliability or signal-to-noise reported here represent conservative values - susceptible of improvement by one or more orders of magnitude with the appropriate engineering.

### 1. Skin Depth at 35 GHz

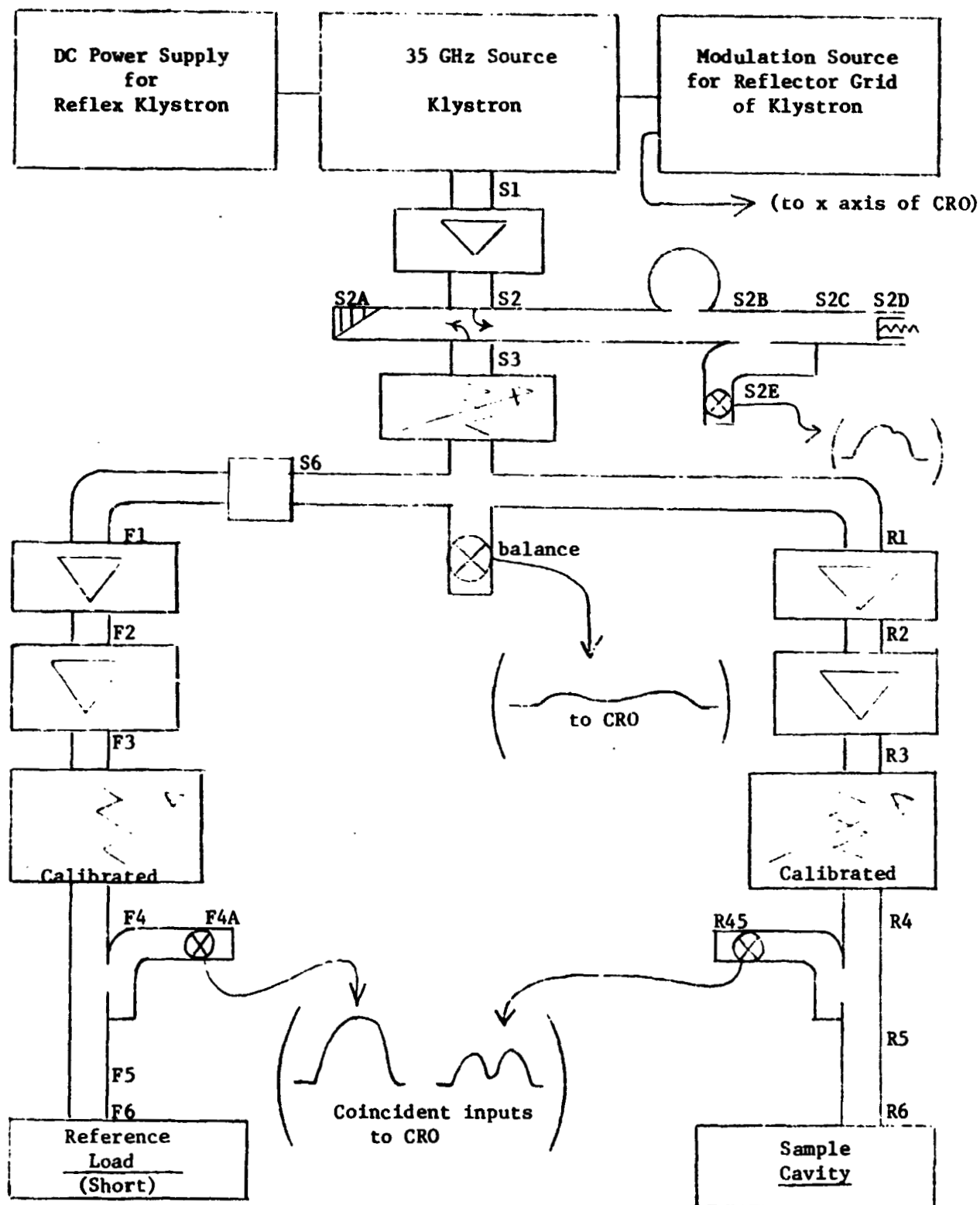
Resistivity near a semiconductor surface is related to the skin depth,  $\delta_s$ , observed at the frequency  $f$  by Eq. (2) above. The sensitivity of the measuring circuit to small changes in  $\delta_s$  depends on the way the semiconductor surface is coupled to the circuit. Two styles of coupling - cavity loading and waveguide termination - were chosen in order to cover a broader range of resistivity values.

#### a. Cavity loading, especially for small single crystals.

The loss introduced into the circuit by a sample of semiconductor surface will be a small fraction of the "no-sample" circuit losses when the product<sup>16</sup> of the surface area times the skin depth  $\delta_s$  is small. This occurs both for small crystals of normal resistivity and for larger sections of large flat surfaces of high resistivity. Cavity loading is preferred because it raises the strength of the electromagnetic field at the semiconductor surface relative to its strength in most other parts of the detecting circuit and this leads to greater sensitivity to losses in the semiconductor surface than without the cavity.

This section describes: 1.) the microwave circuit which was arranged and calibrated to yield signals reliably corresponding to absolute cavity parameters and 2.) the type of substitutional cavity loading used. Our analyses for converting cavity parameters into values of skin depth of the semiconductor sample are given in the appendices.

1.) The double-arm 35 GHz circuit used. In Fig. 1, the 35 GHz double arm reflectometer is shown schematically with a reflective



ORIGINAL PAGE IS  
OF POOR QUALITY

Fig. 1. Schematic of 35 GHz Double-Arm Reflectometer

TABLE 1

## COMPONENTS USED IN 35 GHz DEMONSTRATION DOUBLE ARM REFLECTOMETER

<u>Label on Figure 1</u>	<u>Item - Function</u>	<u>Specific Type Mfg. - Model No.</u>
	DC Power Supply	NARDA Model 438
	Modulation Source	NARDA 438
Kylstron	Source of 35 GHz Power	Varian VA-97
S1	Isolator (20 db)	TRG A110-95
S2	Directional Coupler (20 db)	Microline 405A
S2A	Matched Load	Part of S2
S2B	Frequency Meter (Tunable Cavity)	H-P R532A
S2C	Directional Coupler (20db)	Microline 429A
S2D	Waveguide Short (Adjustable)	
S2E	Crystal Holder (Tunable)	Demornay Bonardi
S3	Attenuator (Variable)	Lieco 10V1-26
S4	Magic Tee (4 arm)	Demornay Bonardi
S5	Crystal Holder (Tunable)	
S6	Slide Screw Tuner (for balancing Tee S4)	Demornay Bonardi DBD 919
Front Arm		
F1	Isolator (20 db)	Uniline by Cascade Res.
F2	Isolator (20 db)	TRG A110-39
F3	Calibrated Attenuator of Rotating Vane	H-P R382A
F4	Directional Coupler (10 db- broad band)	TRG A561-1C



Table 1 - (Contd)

<u>Label on Figure 1</u>	<u>Item - Function</u>	<u>Specific Type Mfg. - Model No.</u>
F4A	Tunable Crystal Holder (Containing MA494 crystal selected for having power response (I vs. $P_{in}$ ) similar to the crystal in holder R4A)	Microwave Associates Mod. 5130
F5	Waveguide (connection between reference short and front directional coupler)	UAH - usually included a piece of stainless steel waveguide 20 $\lambda_g$ long 8.448 $\pm$ .002 inch, having 2.2 db round trip loss.
F6	Reference Load - A low loss, $\lambda_g/4$ short arranged by F5 to be the same path length from the front F4 directional coupler as the sample cavity iris is from the rear R4 directional coupler.	UAH - Dwg available.
Rear Arm - Each corresponding item is the same model as in the front arm, except:		
R6	Sample Cavity - usually TE <sub>015</sub> mode rectangular cavity incor- porating: 1. Special sample holder 2. Fixed (but demountable) coupling iris.	UAH - assembled copper pieces, dwgs available.

sample cavity on one arm and a high quality waveguide short on the reference arm. The list of the components used in this circuit is given in Table 1 and points to the identical characteristics of the two arms, including matching the power response of the detecting crystals over the frequency range of some 100 MHz width.

Frequency modulation over one klystron mode presented the two crystal outputs simultaneously on a double input scope. By careful calibration, including frequent substitutions of standard metal samples, the desired cavity reflection coefficient change was determined from the change in the calibrated attenuator  $R_3$  required in order to rematch the CRO traces upon each substitution of the sample. The formulae used are given separately in an appendix for the different types of cavity loading.

2.) Types of cavity loading used. Rectangular  $TE_{01p}$  mode cavities were constructed of milled sections of copper bolted together with one section carrying a fixed circular iris<sup>17</sup> in a thin (.020 inch) wall and one section mounting the sample. With the minimum milled radius of inside corners at .016 inch, we found it necessary to have the sample cavity at least five half wavelengths ( $p = 5$ ) long. Greater sensitivity will accrue to sample cavities formed to permit  $p = 2$ . The need for the fixed iris is evident in the formulae in the appendices, permitting cavity changes upon substitution to be related solely to the sample. The original and still useful reference on iris design is MIT Rad Lab Report 43-22 by H. A. Bethe.<sup>17</sup>

Loading of the cavity by a semiconductor surface was done in one of two ways: by replacing the end wall or by adding surface in the transverse plane at one-half wavelength from the end wall. Flat GaAs surfaces were held at the end of the rectangular  $TE_{01p}$  mode and leakage losses were held to a satisfactorily low level by appropriate choke joints. Clearly an improved design with much lower coupling losses would involve a sample cavity in the shape of a right circular cylinder<sup>4</sup> and the semiconductor flat as its end wall or central portion thereof.

Thin semiconductor (GeSe) flakes whose other two dimensions were under 7 and 3.5 mm, respectively, were suspended between two strips of plastic (sample holder) at one half wavelength up. Fig. 2 indicates the microwave magnetic field pattern in the last two half wavelengths in the  $TE_{01}$  mode, outlined by the cavity walls and intercepted by the sample at one-half wavelength up where the microwave magnetic field is parallel to the largest faces of the thin flakes. Our tests indicated no appreciable mode distortion by the thin GeSe flakes used so the observed changes in  $Q$  were related to losses on the GeSe surfaces. We assumed these losses were conduction losses within the GeSe and calculated the appropriate skin depths after integrating over the flake area.<sup>16</sup>

b. Waveguide termination. For mapping flat semiconductor surfaces having low resistivity (below about 1 ohm-cm), the simple termination of a waveguide by the sample surface being held across the guide opening will give reasonable sensitivity. One expects, for example, a factor of 4 in standing wave ratio at 35 GHz for a factor of ten in resistivity

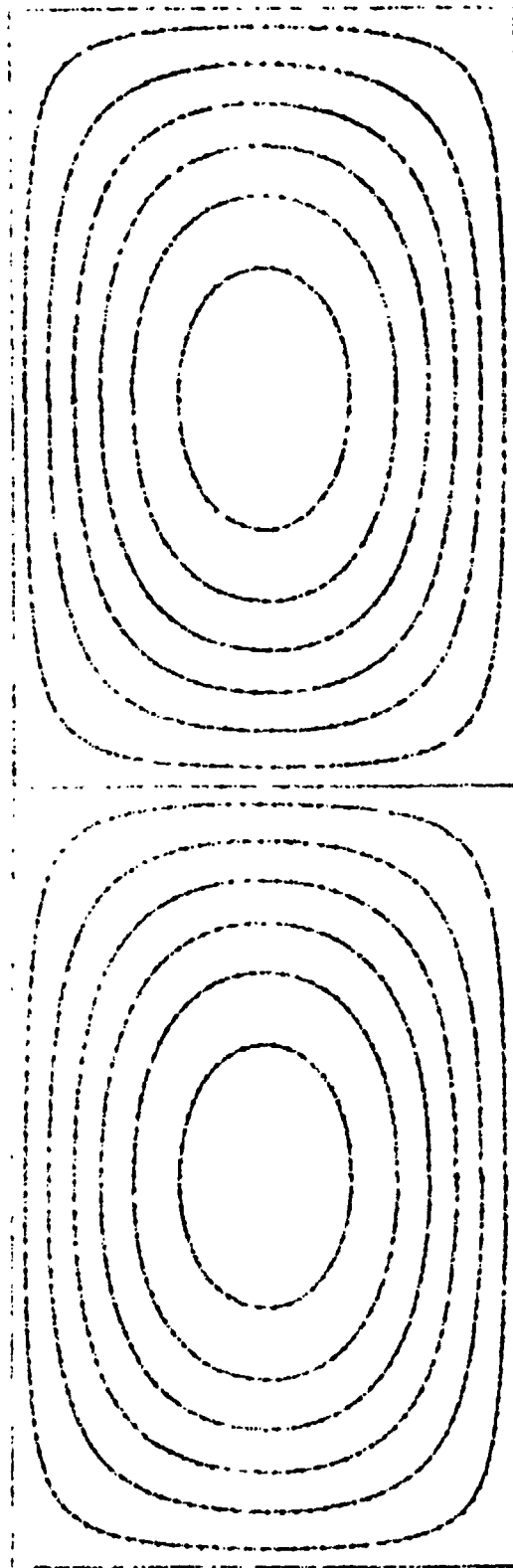


Fig. 2. One wavelength of the pattern of microwave magnetic field in rectangular  $TE_{01p}$  mode sample cavity as viewed through a broad face.

Sample Plane

Credit: George R. Smith for this accurate representation of the H-pattern and its generation using an HP 9100 Plotter.

End Wall

according to Lundmayer et al.<sup>15</sup> Again, the effects of leakage on variations in the choke coupling can be effectively eliminated for surfaces flat to within one or two degrees over small sampling areas by using circular guide.

Our preliminary tests confirmed the suitability of choke coupling for mapping GaAs flats with as cut surfaces when held as rectangular waveguide terminations.

## 2. Fluorescence from Electron-Hole Recombination

The recent surge in the semiconductor market for light-emitting diodes for optical couplers and lasers as well as for displays has placed considerable additional premium on improving semiconductor crystal quality in the category of gallium-aluminum phosphide-arsenide. Of special interest is the spectrum of luminescence and the density of donor sites. Our fluorescence method of surface characterization dealt directly with these two properties of a GaAs surface on a resolution scale of a fraction of a mm. Fluorescence measurements are inherently capable of characterization on a resolution scale of the dimensions of either the carrier mean-free path or the diffraction limit of the optical system used to excite the carriers - whichever is the larger for the sample in question.

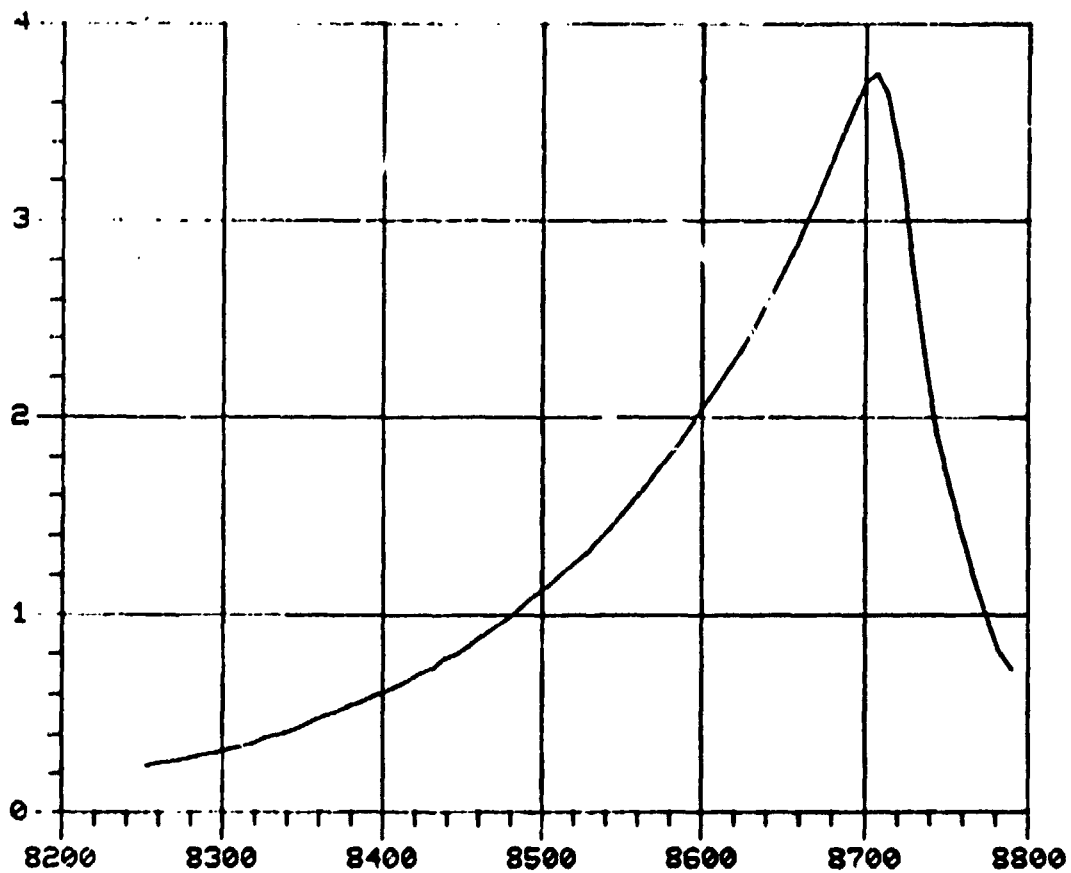
The strong market for microwave generation and light modulation in Ga-Al-P-As semiconductors merits more direct measurements of carrier lifetimes than by standard fluorescence. We suggest optically-pumped cyclotron resonance should do well.

Our approach was to use the photoluminescence reported<sup>10,12</sup> for gallium arsenide crystals at low temperatures, where the different kinds of donor centers can be resolved in the emission spectra, in interpreting the room temperature luminescence. We pumped a spot about 0.5 mm diameter on a GaAs surface with a laser beam, delivering some 20 milliwatts at 632.8 nm, and collected the emission over a solid angle of approximately 0.3 steradians not including the angle of specular reflection. After passing through a double monochromator at low resolution (1 mm slits), a narrow band of the emitted light is collected on a red sensitive photomultiplier tube (C3893) and the output displayed on a chart recorder as the monochromator wavelength is scanned from 8200 Å to 8800 Å in about 10 minutes.

The principal features of a chart record of the room temperature fluorescence from a single spot on GaAs are: the total intensity, the wavelength of the maximum intensity and the widths of the low energy tail and of the high energy side. A typical spectrum from GaAs is displayed in Fig. 3. Mapping is done effectively by repositioning the GaAs surface for each spot of interest, with micrometer drives.

Credit goes to Mr. James M. Rowe for arranging, calibrating and tuning up the apparatus to the state where we could scan a set of spots and then get reproducibility in total intensity to within ten percent. The wavelength for the peak indicates the kind of e-h recombination center active (usually a donor site). When the tails on either side are narrow, the total intensity is related directly to the number of these donor centers being excited by the pump light. With the focus and power of the pump light held constant, the total emitted intensity is proportional to the local concentration of donors.

Identification of the type of donor site by correlation with the value of the wavelength at the peak of each spectrum will require correction of the PMT output to one proportional to the number of photons collected. J. M. Rowe used a standard lamp to calibrate the PMT output and with the aid of Karla Dalton prepared an effective computer program for carrying out this correction procedure. The program's flow chart is given in the last appendix. The output of the program in its present form is automatic graphing of: the input spectral data, the corrected PMT output vs. wavelength, and the corrected PMT output vs. energy of the emitted photons. Examples of these automatic graphs for the emission spectrum of one spot on a GaAs as-cut surface at room temperature are displayed in Figures 3, 4 and 5.

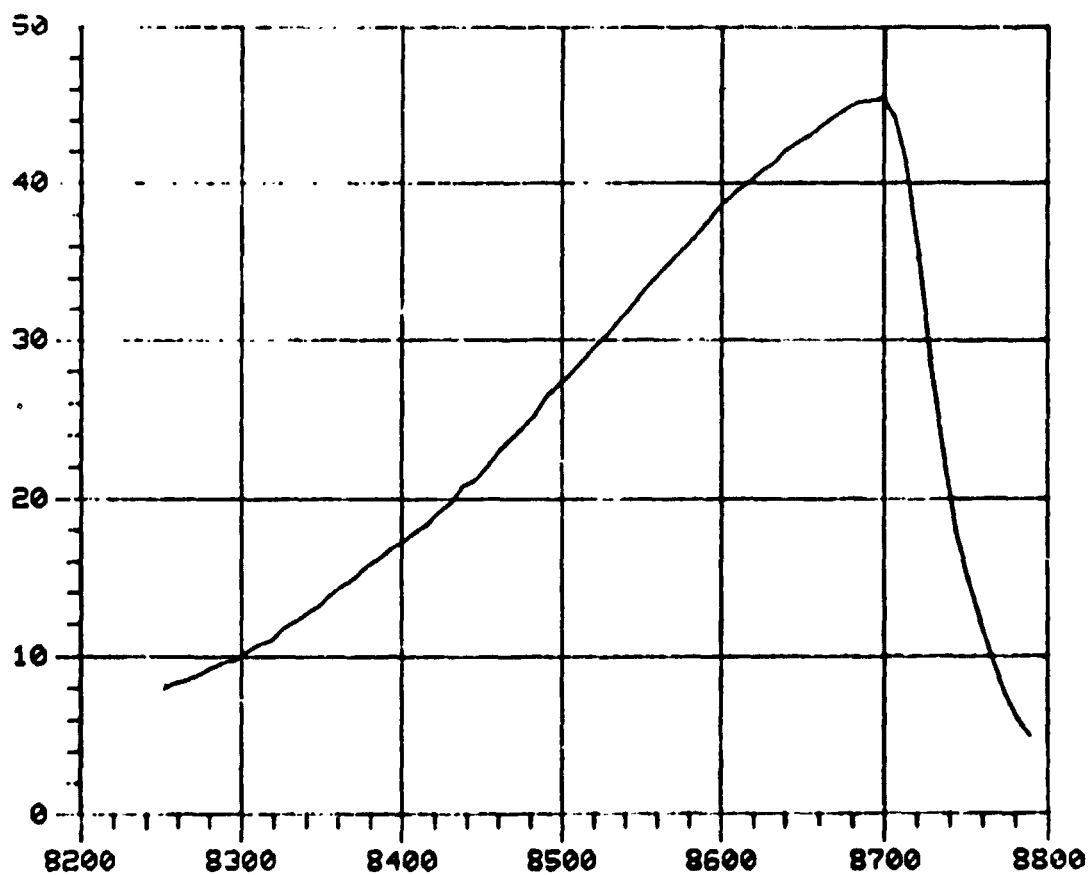


CORRECTED PMT OUTPUT, AMPLITUDE VS WAVELENGTH

756-1120 C DATA SOURCE

Fig. 3. Photoluminescence Observed from GaAs.

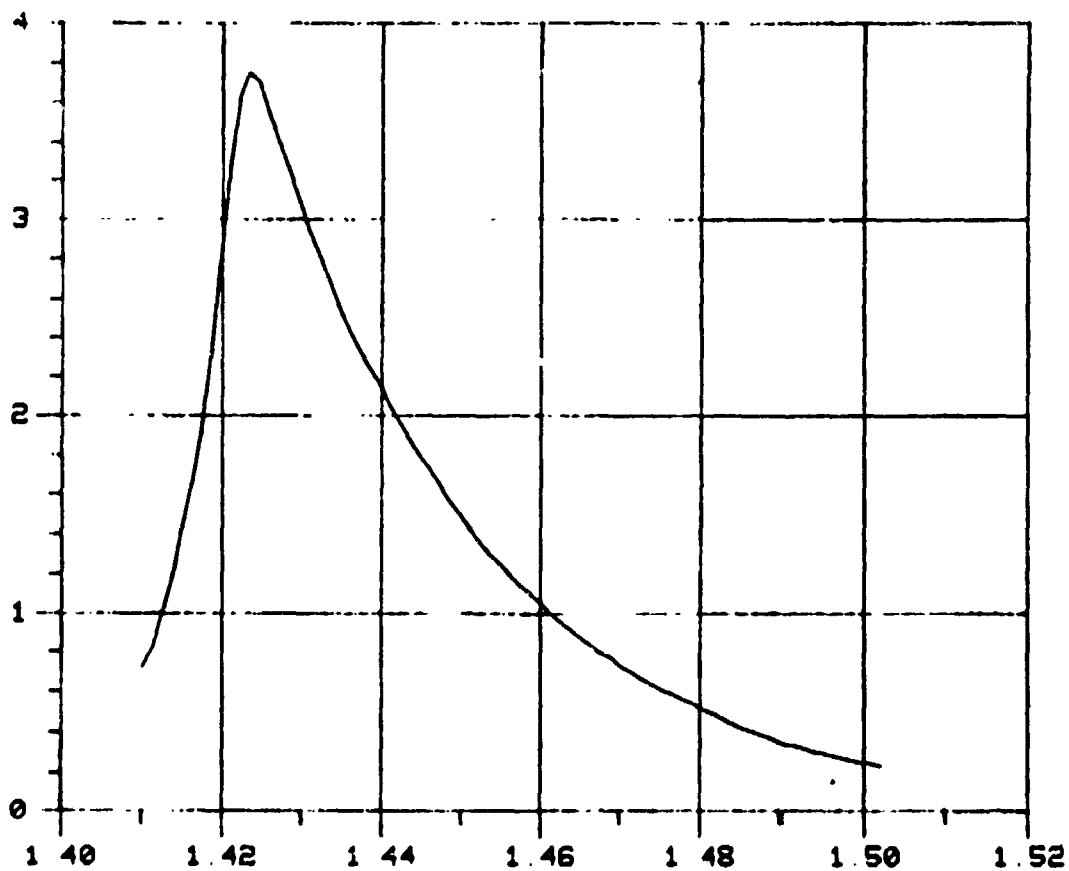




PMT OUTPUT. AMPLITUDE VS WAVELENGTH

7361120 C DATA SOURCE

Fig. 4. PL from GaAs.



CORRECTED PMT OUTPUT, AMPLITUDE VS ENERGY (eV)

7361120 (2) DATA SOURCE

Fig. 5. PL from GaAs

### III. RESULTS - DEMONSTRATION OF SENSITIVITY OF NONCONTACTING TECHNIQUES

In direct assistance to the NASA space processing program, this study has developed three noncontacting techniques into successful laboratory procedures for the nondestructive electrical characterization of single crystal semiconducting surfaces. Time permitted only preliminary measurements with each of the three procedures, but an indication of their sensitivity is given in the precision stated below for the measured characteristics. Values obtained for the skin depth at 35 GHz and 300 °K in selected GaAs surfaces are listed, following a description of the skin effect found in a few of the GeSe flakes grown on Skylab missions.

#### A. Skin Depths in GeSe Flakes at 35 GHz

The high quality single crystal flakes produced by vapor transport in ampules in Skylab's multipurpose furnace were thin platelets a few square millimeters in area. We developed the procedure described above for loading a room temperature 35 GHz copper cavity with one GeSe flake at a time so that the cavity reflections could be interpreted to yield the ratio of the skin depth averaged over each flake to the skin depth of the copper walls of the cavity. A precision measurement of cavity Q would then yield a precise value of the average  $\delta$  in each flake.

In our preliminary laboratory operation, we obtained introductory information on several specific questions about the use of 35 GHz radiation to characterize small semiconducting crystals. First, our procedures are capable of characterizing without damage the skin depth of each of a large number of crystals per day. Secondly, centering of the sample was judged visually and remained one of the principal sources of fluctuation in our

observed values. Except for more difficult sample handling, the same cavity loading procedures work at liquid nitrogen temperature with the expectation of even better sensitivity. Some care must be exercised in the choice of materials for holding the sample flakes in place within the copper cavity, but polyethylene film appears to serve well at our present sensitivity. It should also be noted that the surface currents induced in the flat platelet when properly mounted in cavity are all in one direction, the same on both sides of the platelet, permitting thereby a search for anisotropy in the resistivity of each semiconductor crystal.

One operational aspect was tested - namely the skill level required for carrying out the cavity loading observations on GeSe at 35 GHz. After the selected GeSe flakes had been measured by a highly experienced operator (#1) to be certain that the loading procedure functioned satisfactorily, in the reflection cavity (A), verbal instructions were given to a new technician (operator 2) who had experience with ham radio but none with microwaves. Operator 2 then surveyed the same samples in cavity A and again in a modified cavity (B) in which the end of the cavity had been modified to permit transmission measurements.

The room temperature results are summarized in Table 2. The relative Q factors ( $Q_s$ ) of each sample to that ( $Q_w$ ) of the cavity walls are listed as an aid to later evaluation of this cavity loading technique. Pertinent details are listed in the appendices.

Inspection of Table 2 shows how the GeSe skin depths vary between these five crystals by a factor of 6. The corresponding range of equivalent

TABLE 2

RELATIVE Q AND SKIN DEPTHS FOR GeSe FLAKES  
AT 35 GHz AND ROOM TEMPERATURE

Crystal		$Q_{\text{wall}}/Q_{\text{sample}}$				$\delta_s/\delta_{\text{cu}}$	$\delta_s$
Label	Total Area mm <sup>2</sup>	Combinations Used**			Mean Value $Q_w/Q_s$		micron*
		A,1	A,2	B,2			
3B-FS	1.86	0.119 ± .015 from 3*	0.134 ± .013 from 2*	0.159 ± .015 from 2*	0.137 ± .014	16.2 ± 1.6	5.7
3B-F6	0.96	0.018 ± .003 from 6*	0.014 ± .004 from 2*	0.015 ± .005 from 2*	0.016 ± .004	3.7 ± 0.9	1.3
3A-F1	1.11	- tried 2*	- tried 2*	0.015 ± .005 from 2*	0.015 ± .005	2.7 ± 0.9	0.9
3A-F2	0.89	0.016 ± .005 from 4	0.038** ± .008 from 2*	0.020 ± .005 from 2*	0.025 ± .010	6.0 ± 2.4	2.1
RPI-A (H1)	6.5	0.150 ± .005 from 2*	0.149 ± .005 from 2*	0.139 ± .02 from 1*	0.149 ± .005	2.9 ± 0.1	1.03

\* Number of independent substitutions of sample into cavity.

\*\* First set for operator 2

● Calculated with  $\delta_{\text{cu}} = 0.15$  micron

●● Combinations used were Cavity A by operator 1, Cavity A by operator 2, and Cavity B by operator 2.

bulk resistivities is a factor of 36. The uncertainties observed would have been reduced - probably to  $\pm .003$  in  $Q_w/Q_s$  - by using ten or so independent substitutions of the sample during each run.

The data represented in Table 2 also confirm the soundness of our theoretical analysis in regard to sample configuration. The absolute values of the skin depth,  $\delta_s$ , listed in the last column of Table 2 confirm the validity of the flakes being thick compared to their own skin depth so that the power loss in a flake is the same kind of product<sup>16</sup> of surface integrals of  $H_T^2$  as for the copper walls. Secondly, the flakes were thin enough to permit using the undistorted mode values for  $H_T$  across the sample.

A final note about sensitivity of this cavity loading substitution procedure is that when a flake of ultra high purity aluminum foil was the substituted load, the minimum detectable area was about  $3 \text{ mm}^2$  of Al. As the resistivity of the semiconductor flake is increased, our cavity loading signals become larger, indicating the capability of characterizing smaller flakes or of obtaining more precision. For example, the observed standard deviations listed in Table 2 give:  $\pm 3\%$  for a  $7/2 \text{ mm}^2$  flake having  $\delta_s = 1$  and  $\pm 10\%$  for a  $1.9/2 \text{ mm}^2$  flake having  $\delta_s = 6$  micron.

#### B. Skin Depths in GaAs Flats at 35 GHz

High quality boules of GaAs grown by M. Rubinstein of Westinghouse Research and Development Center in preparation for the M555 Experiment on Skylab had been sliced for characterization. Several cut faces, termed flats, were mapped for skin depth at 35 GHz using a resolution element of  $4 \times 7 \text{ mm}$  as the rectangular  $TE_{01p}$  cavity end wall in the procedure mentioned

above. Cavity reflections were interpreted in accord with the analysis outlined in the appendices in terms of the skin depth  $\delta_s$  in the GaAs. Some of the average values found for  $\delta_s$  are listed in Table 3, taking  $\delta_{Cu} = 0.35$  micron.

TABLE 3  
CLASSICAL SKIN DEPTH

Sample	$Cs^1(p=13) \quad \delta_s(\text{mm})$	$Cs^2(p=7) \quad \delta_s(\text{mm})$
W-2	$(1.178 \pm 1.2) \times 10^{-2}$	(--)*
W-3	$(2.053 \pm 2.66) \times 10^{-2}$	$(1.014 \pm 0.3) \times 10^{-2}$
E-2	$(2.423 \pm 2.07) \times 10^{-2}$	$(2.056 \pm 0.6) \times 10^{-2}$

\*A value for  $\delta_s$  was not obtainable from the data.

The uncertainty in the measured values was much larger than the limiting uncertainty due solely to the ability to read the equipment accurately. Evidently the variations in resistivity between the areas being sampled are comparable to but not much larger than the variations in coupling as an end wall in one positioning of the sample. Precision improvement of almost one order of magnitude is expected in the same procedure when the shape of the sample cavity is a right circular cylinder and the resolution element of the semiconductor flat is the circular end wall or a concentric circular portion.

The actual values obtained, however, from the two cavities,  $Cs^1$  and  $Cs^2$ , for the classical skin depth of the samples, are in good agreement. Clearly the shorter cavity produces higher precision as expected. Again, when

machining or etching techniques are used that permit sharper inside corners (than  $r = 0.016$  inches), then  $p = 2$  cavities can be used and precision will be improved over the  $p = 7$  cavity.

### C. Mapping of GaAs Single Crystals by Fluorescence

Photo-induced luminescence of the electron-hole recombination radiation has been observed by J. M. Rowe from cut faces (flats) of several high quality GaAs crystals. The technique outlined above permits mapping by repositioning the flat in the plane of its irradiated face by micrometer drives. A spectrum is scanned for each position of the laser beam spot. Fig. 3 shows, with the aid of automatic graphing on the UNIVAC 1108 computer, the typical spectrum emitted from a spot on the face of a crystal doped with  $\sim 10^{17}$  donors/cm<sup>3</sup>. The total intensity (proportional to the donor concentration<sup>10</sup>) was found to be reproducible to  $\pm 10\%$  for 10 minute spectral scans of the same spot location and relocations. Observed variations across a sample were occasionally 50% and were, therefore, attributable to donor concentration variations, but no direct corroboration of the same surface profile was available at the time of the fluorescence scans. The automatic correction of PMT output via standard lamp calibration is discussed in an appendix and the corrected results plotted in Fig. 4 and 5 of the spectrum of Fig. 3.

Uniformity of surface roughness probably represents the ultimate limit to the precision of this high resolution method of characterizing the donor concentration. Changes in the type of donor will shift the peak of the spectrum. For example, nitrogen donors are a few millielectron volts below the band edge at  $E_g \sim 1.43$  eV. No attempt was made to correlate peak position



with active donor types, but it is clear<sup>12</sup> that lower temperatures permit complete resolution of each type of donor traps.

#### IV. SUMMARY AND PROGNOSIS

The results of this study of nondestructive methods of electrical characterization of the higher quality single crystals of semiconductor grown in the microgravity of space environment include both the demonstration of successful methods - two microwave and one optical - and the measurement of the resistivity of a few of the small GeSe crystals grown by vapor deposition on Skylab flights. The observed sensitivity of the two microwave methods - one for mapping large flat semiconductor surfaces and one for observing the whole surface of small thin flat crystals - is reported for a limited range of resistivity. The relative skin depth values for the GeSe flakes studied by a sample substitution procedure were found to have standard deviations around 10% for rather small crystals. The absolute skin depth values could readily have had the same precision if we had made the frequency measurements which lead to the absolute  $Q_L$  values.

Our prognosis is optimistic, namely that these room temperature microwave techniques can be carried out nondestructively using light weight apparatus with adequate precision for measuring uniformity of resistivity of high quality semiconductor surfaces. Furthermore, related noncontacting techniques such as cyclotron resonance and photoinduced microwave conductivity show promise of being useful sources of electrical characteristics of the high quality semiconductors to be grown in space.

The observed reproducibility of the fluorescence of room temperature GaAs under irradiation at 632.8 nm of some 10% in donor density for our slow scan technique also shows promise for NASA applications. Actually, with the large expansion in the light emitting diode production in the

past several years, the LED manufacturers very likely have a similar optical scanning technique in regular use by now.

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## APPENDIX A

## ASSUMPTIONS FOR EQUATIONS IN APPENDICES B AND C

If all of the microwave energy is stored in the rectangular cavity in only one  $TE_{01p}$  resonant mode, it is possible to find a point,  $z_0$ , such that the electric field is 0, independent of the x and y positions and time. Thus, we can insert a very thin conducting sample and a sample holder, made of a dielectric, at that point, with its thin dimension parallel to the z-axis without changing the electric field. If  $\mu_s = \mu_H = \mu_0$ , where  $\mu_s$  is the permeability of the sample and  $\mu_H$  is the permeability of the holder, then we do not change the magnetic field and thus the energy stored in the empty cavity is the same as that of the cavity with sample holder with or without the sample.

If the electric and magnetic fields are not altered within the cavity by the addition of the sample holder and sample, then the intrinsic impedance of the cavity has not changed. Thus, the energy radiated out the coupling iris, back down the waveguide should not change. This means that, for cases (A) through (C) the value of  $\left(\frac{1}{Q_{c,1}}\right)$  should be the same. Similarly, if, by adding the sample and sample holder to the empty cavity, we do not change the current density in the cavity walls, then the ohmic losses due to the walls is the same in cases (A) through (C). This can be done, for example, by making the sample holder out of a lossless dielectric so that the conducting sample is isolated from the walls of the cavity. A problem can arise, however, in the manner in which the sample holder and sample are inserted into the cavity. In this particular process, a slot was cut into the side of the cavity. This means that some power was

radiated out of the cavity. This loss is usually neglected in the total loss of power due to the walls of the cavity. We assume that adding the slot did not change the electric and magnetic field configuration in the cavity, and, as we are in a  $TE_{01p}$  mode, this means that at  $Z_0$ , where the slot was made, the tangential electric and tangential magnetic fields are both zero. Thus the amount of energy radiated out of the cavity is only a small part of the total wall losses. Thus, altering this amount of radiated energy, such as is caused by inserting the dielectric sample holder into the slot, does not significantly alter the total energy loss due to the walls of the cavity. Hence the value of  $Q_W$  does not change for cases (A) through (C). A similar argument shows that the value of  $Q_W$  is also a constant for cases (D) through (F).

If we assume that the addition of the second coupling iris does not change the resonant mode electric and magnetic field of the single-line cavity-coupling system, then  $(Q_W)$  does not change from case (A) through (F) as the total energy stored will be the same, and the change in the energy lost due to the walls is not greatly changed by the loss of the surface area, due to the second coupling iris, from  $\int_S H_T^2 ds$ , which is proportional to the ohmic energy loss per cycle due to the walls of the cavity,<sup>(1)</sup> where  $H_T$  is the amplitude of the magnetic field tangent to the walls of the cavity and the integral is taken over the total surface of cavity walls. The assumption that the addition of the second coupling iris does not change the resonant mode is justified in the equipment and cavities used, by the fact that the resonant frequency shifted by less than 0.1% from the single-line cavity-coupling system to the two-line cavity-coupling system.

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(1) See E. U. Condon, Rev. Mod. Phys. 14, P-341 (1942).

Finally, if the addition of a second coupling iris does not change the electric and magnetic fields in the cavity, then the value of  $Q_{c, 1}$  is the same in cases (A) through (C) and cases (D) through (F). Thus, the values of  $Q_W$ ,  $Q_{c, 1}$ ,  $Q_S$ , and  $Q_H$  are the same for cases (A) through (F). Hence, equation (18) is justified. It also means that the values of  $\beta_{1, MT}$  and  $\beta_{2, MT}$  are the same in both the single-line and two-line cavity-coupling system, so that we can measure both values by using the single-line cavity-coupling system and use these values in an equation that is true for a two-line cavity-coupling system. Thus we can obtain a justifiable value for  $Q_S$  using equations (19), (20), and (17) or (17A).

If we still assume that all the microwave energy of the rectangular cavity is stored in only one  $TE_{01p}$  mode and that the electric and magnetic fields in this mode are the same for the coupled cavity as for the uncoupled cavity, then the total energy stored in the empty cavity is  $\frac{1}{2} \int_V \mu_0 H^2 dv$  where  $H$  is the magnitude of the magnetic field and the integral is taken over the total volume of the cavity.<sup>2</sup> If we insert our sample and sample holder in the cavity at the point  $Z$  such that the electric field is zero, then the energy stored in the cavity with the sample and sample holder is the same as the empty cavity.

As the sample is conducting, it will create an ohmic power loss that will increase the energy loss per cycle in the cavity. The energy loss per cycle in the sample is given by  $\frac{\delta_s \pi \nu}{2} \int_S H_T^2 ds$  where  $\delta_s$  is the skin depth of the sample and  $H_T$  is the magnitude of the magnetic field tangent to the surface of the sample  $A$  and the integral is taken over the total surface of the sample.

2. See Fig. 2 of this report for the shape of the  $H$  pattern.



$$As Q_s = \frac{2\pi(\text{Energy Stored in the Cavity})}{(\text{Energy Lost in the sample in one cycle})}$$

and we have already assumed  $\mu_s = \mu_0$  we get,

$$Q_s = \frac{2 \int_v H^2 dv}{\delta_s \int_s H_T^2 ds} \quad (27)$$

Using the solutions for the electric and magnetic fields in the  $TE_{01p}$  mode<sup>4</sup> and assuming that the sample is a rectangular parallelepiped, we can perform the volume and surface integrals above, remembering that the sample is at  $z_0$ , we get,

$$Q_s = \frac{\pi AC [B^2 p^2 + C^2]}{4\delta_s B^2 p^2 (\Delta x) \left[ \frac{\pi}{2B} \Delta y - \frac{1}{4} \sin\left(\frac{2\pi}{B} y_2\right) + \frac{1}{4} \sin\left(\frac{2\pi}{B} y_1\right) \right]} \quad (28)$$

Where A is the length of the cavity along the x-axis, B is the length along the y-axis, C is the length along the z-axis,  $\Delta x$  is the length of the sample along the x-axis  $\Delta y$  is its length along the y-axis and  $y_1$  and  $y_2$  are the y positions of the sample in the cavity. This result also assumes that  $\Delta z$  of the sample is so small that we can ignore the surface integral over those portions of the surface of the sample that involve  $\Delta z$ . This is justified in all the GeSe flakes measured.

Other shapes will produce other solutions, however, most shapes can be approximated by a rectangular parallelepiped or a sum of such shapes, in which case, the above equation will still hold. Otherwise, other

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4. See Technique of Microwave Measurements, Vol II, edited by C. G. Montgomery, P. 295, McGraw-Hill (1947).

coordinate systems and techniques of integration will have to be used. Also, note that the factor of 4 appears in equation (28), rather than a factor of 2, to account for the loss in both sides of the sample, so that, if a sum of rectangular parallelepipeds is to be used as the approximation, this factor can be changed to 2 and the sum carried over both sides independently.

Finally, if we again have a rectangular parallelepiped in which we can ignore the  $\Delta z$  terms and further, that  $\Delta y$  is so small that  $\sin^2\left(\frac{\pi}{B}\left(y_1 + \frac{\Delta y}{2}\right)\right) = \sin^2\left(\frac{\pi}{B} y_1\right)$ , then we arrive at a good approximation for  $Q_s$  by assuming that  $H_T^2$  is a constant over the total surface area of the sample; that is

$$Q_s = \frac{AC[B^2 p^2 + C^2]}{4 \delta_s B p^2 \sin^2\left(\frac{\pi}{B}\left(y_1 + \frac{\Delta y}{2}\right)\right) \Delta x \Delta y} \quad (29)$$

The basic assumption in the choke joint cavity system is that the resonant electric and magnetic fields are the same in all four types of cavities. The same reasoning applies in these sets of cavities in transferring from the single line cavity-coupling system (cavities X, Y, and Z) to the two-line cavity-coupling system (cavity T). Any difference in microwave energy losses, due to the choke joint, between the Z cavity and the X cavity is taken into account by the  $Q_{R, C, H}$  term. Finally, the same reasoning is used in calculating the classical skin depth,  $\delta_s$ , of the sample from  $Q_{W, S}$ , as was used in the preceding cavity system, keeping in mind that now the sample makes up one complete end wall of the cavity.

## APPENDIX B

BASIS FOR LOSS CALCULATIONS FOR  $\text{Cu TE}_{01p}$  RECTANGULAR CAVITIES  
FOR  $f$  LARGE ENOUGH TO SUSTAIN NORMAL MODE CONFIGURATIONS

Terms for Single Line Cavity-Coupling System:

- $P = 2C/\lambda_g$   
 $c =$  Cavity dimension along  $z$  axis  
 $\lambda_g =$  Wave length in the cavity at resonance  
 $Q = 2\pi$  (energy stored/energy dissipated per cycle)  
 $Q_{L,MT} =$  Loaded  $Q$  of empty cavity  
 $Q_{L,0} =$  Loaded  $Q$  of cavity with sample holder but without sample  
 $Q_{L,s} =$  Loaded  $Q$  of cavity with sample holder containing sample  
 $Q_W =$   $Q$  of Cu walls of cavity  
 $Q_{c,1} =$   $Q$  of 1st coupling iris  
 $Q_H =$   $Q$  of sample holder  
 $Q_s =$   $Q$  of sample  
 $Q_{u,0} =$   $Q$  of Cu walls and sample holder  
 $Q_{u,s} =$   $Q$  of walls, sample holder and sample  
 $\beta =$  Geometrical coupling parameter which is a constant for a given iris and cavity mode. Each value of  $\beta$  is obtained from a measured reflected power ratio  $\Gamma$ .  
 $\Gamma =$  Power ratio (Power reflected/Power incident at plane of cavity iris)  
 $\beta_{1,0} =$  Coupling parameter of 1st coupling iris with empty sample holder  
 $\beta_{1,s} =$  Coupling parameter of 1st coupling iris with sample  
 $\beta_{1,MT} =$  Coupling parameter of 1st coupling iris with empty cavity

Terms for Two-Line Cavity Coupling System:

$Q_{L,MT,T}$	=	Loaded Q of empty transmission cavity
$Q_{L,O,T}$	=	Loaded Q of cavity with empty sample holder
$Q_{L,s,T}$	=	Loaded Q of cavity with sample holder containing sample
$Q_{c,2}$	=	Q of 2nd coupling iris
$Q_s$	=	Q of sample
$Q_H$	=	Q of sample holder
$Q_{u,O,T}$	=	Q of Cu walls and sample holder
$Q_{u,O,s}$	=	Q of walls, sample holder and sample
$Q_{c,2}$	=	Q of 2nd coupling iris
$\beta_{2,MT}$	=	Coupling parameter of 2nd coupling iris with empty cavity
$\beta_{2,O}$	=	Coupling parameter of 2nd coupling iris with empty sample holder
$\beta_{2,s}$	=	Coupling parameter of 2nd coupling iris with sample holder
$f_o$	=	Resonant frequency of empty cavity
$\Delta f(3db)$	=	Width of power resonance curve at half-peak values

The following equations are applicable when the loss mechanisms are independent and the microwave energy is stored in only one mode.

Basic Equations for Single-Line Cavity Coupling System:

A. For the Empty Cavity

$$1. \quad \frac{1}{Q_{L,MT}} = \frac{1}{Q_{c,1}} + \frac{1}{Q_W}$$

$$2. \quad \beta_{1,MT} = \frac{Q_W}{Q_{c,1}}$$

B. For Cavity with Empty Sample Holder

$$3. \quad \frac{1}{Q_{L,0}} = \frac{1}{Q_{c,1}} + \frac{1}{Q_W} + \frac{1}{Q_H}$$

$$4. \quad \frac{1}{Q_{u,0}} = \frac{1}{Q_W} + \frac{1}{Q_H}$$

$$5. \quad \beta_{1,0} = \frac{Q_{u,0}}{Q_{c,1}}$$

C. For Cavity with Sample Holder and Sample

$$6. \quad \frac{1}{Q_{L,s}} = \frac{1}{Q_{c,1}} + \frac{1}{Q_W} + \frac{1}{Q_H} + \frac{1}{Q_s}$$

$$7. \quad \frac{1}{Q_{u,s}} = \frac{1}{Q_W} + \frac{1}{Q_H} + \frac{1}{Q_s}$$

$$8. \quad \beta_{1,s} = \frac{Q_{u,s}}{Q_{c,1}}$$

## Basic Equations for Single-Line Cavity Coupling System (contd)

For the Empty Cavity

$$9. \frac{1}{Q_{L,MT,T}} = \frac{1}{Q_{c,1}} + \frac{1}{Q_W} + \frac{1}{Q_{c,2}}$$

$$10. \beta_{2,MT} = \frac{Q_W}{Q_{c,2}}$$

E. For cavity with empty sample holder

$$11. Q_{L,O,T} = \frac{1}{Q_{c,1}} + \frac{1}{Q_W} + \frac{1}{Q_{c,2}} + \frac{1}{Q_H}$$

$$12. \frac{1}{Q_{u,O,T}} = \frac{1}{Q_W} + \frac{1}{Q_H}$$

$$13. \beta_{2,O} = \frac{Q_{u,O,T}}{Q_{c,2}}$$

F. For cavity with sample

$$14. \frac{1}{Q_{L,s,T}} = \frac{1}{Q_{c,1}} + \frac{1}{Q_W} + \frac{1}{Q_H} + \frac{1}{Q_s} + \frac{1}{Q_{c,2}}$$

$$15. \frac{1}{Q_{u,s,T}} = \frac{1}{Q_W} + \frac{1}{Q_H} + \frac{1}{Q_s}$$

$$16. \beta_{2,s} = \frac{Q_{u,s,T}}{Q_{c,2}}$$

G. Equations for calculating  $\beta$ 

$$17. \beta = \frac{\frac{1}{1-\Gamma^2}}{1+\Gamma^2} \quad \text{for } \beta < 1$$

or

$$17A. \beta = \frac{\frac{1}{1+\Gamma^2}}{1-\Gamma^2} \quad \text{for } \beta > 1$$

$\beta$  can be chosen to be overcoupled (Eq. 17A) (or undercoupled (Eq. 17)) by convenience of machining and detector sensitivity.

$\beta$  calculated using equation 17 or 17A is the  $\beta$  of the single-line cavity coupling system. However, as we do not change the normal mode nor the shape of the iris, it, therefore, has the same value as in the two-line cavity coupling system.

The fact that the coupling parameter,  $\beta$ , for each case can be calculated from experimental data leads to several important results. First, by using equations (1) through (8), we can relate the sample losses to the cavity wall losses by equation

$$\frac{Q_W}{Q_s} = \frac{\beta_{1,MT} (\beta_{1,0} - \beta_s)}{(\beta_{1,0}) (\beta_{2,MT})} \quad (18)$$

As all the values on the right can be calculated from experimental measurements, a value can thus be assigned to  $\frac{Q_W}{Q_s}$ . From this, a comparison of the  $Q_s$  values of different samples can be made in a given cavity, provided the value of  $Q_W$  does not change. Experimental confirmation that  $Q_W$  had not changed was obtained by finding the value of  $\beta_{1,MT} (= Q_W/Q_{c,1})$  [Eq. 2]) to be the same to within  $\pm 0.4\%$  each time the empty cavity was measured.

Secondly, using equations (2) and (10) we can replace the  $Q$  values in Equation (9) with  $\beta$  values to get equation

$$\frac{1}{Q_{L,MT,T}} = \frac{1}{Q_W} (\beta_{1,MT} + 1 + \beta_{2,MT}) \quad (19)$$

As we can calculate a value for  $(\beta_{1,MT} + 1 + \beta_{2,MT})$ , we can thus get a value of  $Q_W$  from a known value of  $Q_{L,MT,T}$ . Finally, we measure the value of  $Q_{L,MT,T}$  experimentally by using equation

$$1/Q_{L,MT,T} = \Delta f(3db)/f_o \quad (20)$$

We can arrive at a value of  $Q_W$  that is

$$\begin{aligned} Q_W &= \frac{f_o}{\Delta f(3db)} (\beta_{1,MT} + 1 + \beta_{2,MT}) \\ &= Q_{L,MT,T} (\beta_{1,MT} + 1 + \beta_{2,MT}). \end{aligned} \quad (21)$$

Thus, using Equations (18) and (21), we arrive at an equation for  $Q_s$ ,

$$Q_s = \frac{f_o (\beta_{1,MT} + 1 + \beta_{2,MT}) (\beta_{1,0}) (\beta_{1,s})}{\Delta f(3db) (\beta_{1,MT}) (\beta_{1,0} - \beta_{1,s})} \quad (22)$$

dependent totally on measurable quantities.

By using the previously mentioned equations, we can also make measurements on the uncertainty of the quantities  $Q_W/Q_s$  and  $Q_s$

First, by using Equation (15) we find the uncertainty  $\Delta(Q_W/Q_s)$  is given by

$$\begin{aligned} \Delta(Q_W/Q_s) &= \left[ \frac{(\beta_{1,0} - \beta_{1,s})}{(\beta_{1,0}) (\beta_{1,s})} \right] \Delta(\beta_{1,MT}) \\ &+ \left[ \frac{(\beta_{1,MT})}{(\beta_{1,s})^2} \right] \Delta(\beta_{1,0}) \\ &+ \left[ \frac{\beta_{MT}}{(\beta_{1,0})^2} \right] \Delta(\beta_{1,0}) \end{aligned} \quad (23)$$



Secondly, using Equation (19), we find the uncertainty of  $Q_s$  is given by

$$\begin{aligned} \Delta Q_s = & \left[ \frac{(\beta_{1,MT} + 1 + \beta_{2,MT}) (\beta_{1,0}) (\beta_{1,s})}{\Delta f(3db) (\beta_{1,MT}) (\beta_{1,0} - \beta_{1,s})} \right] \Delta(f_o) \\ & + \left[ \frac{f_o (\beta_{1,0}) (\beta_{1,s})}{\Delta f(3db) (\beta_{1,MT}) (\beta_{1,0} - \beta_{1,s})} \right] \Delta(\beta_{2,MT}) \\ & + \left[ \frac{f_o (\beta_{1,MT} + 1 + \beta_{2,MT}) (\beta_{1,0})^2}{\Delta f(3db) (\beta_{1,MT}) (\beta_{1,0} - \beta_{1,s})^2} \right] \Delta(\beta_{1,2}) \\ & + \left[ \frac{f_o (\beta_{1,MT} + 1 + \beta_{2,MT}) (\beta_{1,s})^2}{\Delta f(3db) (\beta_{1,MT}) (\beta_{1,0} - \beta_{1,s})^2} \right] \Delta(\beta_{1,0}) \\ & + \left[ \frac{f_o (\beta_{1,0}) (\beta_{1,s}) (1 + \beta_{2,MT})}{\Delta f(3db) (\beta_{1,0} - \beta_{1,s}) (\beta_{1,MT})^2} \right] \Delta(\beta_{1,MT}) \\ & + \left[ \frac{f_o (\beta_{1,MT} + 1 + \beta_{2,MT}) (\beta_{1,0}) (\beta_{1,s})}{[\Delta f(3db)]^2 (\beta_{1,MT}) (\beta_{1,0} - \beta_{1,s})} \right] \Delta(\Delta f(3db)) \end{aligned} \quad (24)$$

For the case in which the sample is placed in a two-line cavity coupling system, an equation similar to (18) can be developed. In fact, we get

$$\frac{Q_w}{Q_s} = \frac{\beta_{2,MT} (\beta_{2,0} - \beta_{2,s})}{(\beta_{2,0}) (\beta_{2,s})} \quad (25)$$

Then, by using Equations (19), (20), and (25), we can develop equations similar to (21) through (24) which relate  $Q_s$  to the second coupling iris parameters,  $f_o$  and  $\Delta f(3db)$ .

$Q_s$  is defined as,

$$\frac{2\pi(\text{Energy Stored in the Cavity})}{(\text{Energy Dissipated in the Sample in One Cycle})}$$

The energy stored in the cavity is equal to<sup>16</sup>

$$\frac{1}{2} \int_V \mu_0 H^2 dv$$

where  $H$  is the amplitude of the magnetic field. The energy dissipated in the sample is due to ohmic losses and in one cycle is equal to

$$\frac{\delta_s \pi \mu_s}{2} \int_s H_T^2 ds$$

where  $\delta_s$  is the skin depth of the sample,  $\mu_s$  is the permeability of the sample,  $H_T$  is the amplitude of the magnetic field parallel to the sample's surface and the integral is taken over the total surface area of the sample. Thus

$$Q_s = \frac{2\mu_0 \int_V H^2 dv}{\delta_s \mu_s \int_s H_T^2 ds} \quad (26)$$

As the samples are only weakly paramagnetic  $\frac{\mu_0}{\mu_s} = 1$  to within 0.1% Hence we use the following equation for  $Q_s$ ;

$$Q_s = \frac{2 \int_V H^2 dv}{\delta_s \int_s H_T^2 ds} \quad (27)$$

Using the TE mode numbers, we can find the magnetic field configuration within the cavity. Thus, Equation (27) can be solved to a good approximation, as follows:

Our coordinate system is that used in Technique of Microwave Measurements, Vol. 11 of the M.I.T. Radiation Laboratory Series, edited by C. G. Montgomery, McGraw-Hill, Inc. 1947, page 295. The dimensions of our cavity are A, parallel to the x axis, B parallel to the y axis, and C, parallel to the z axis

With the thin sample placed in the cavity such that its corners are located at  $(x_1, y_1, z_0)$ ,  $(x_1, y_2, z_0)$ ,  $(x_2, y_1, z_0)$  and  $(x_2, y_2, z_0)$  where  $z_0 = \Omega \lambda g/2$ ,  $\Omega$  an integer, we arrive at the following solutions to Equation (27) as discussed in Appendix A:

A. If  $(x_2 - x_1) \cdot (y_2 - y_1)$  which is the cross sectional area of the sample is comparable in size to  $(A \cdot B)$  which is the cross sectional area of the cavity, then

$$Q_s = \frac{\pi AC [B^2 p^2 + C^2]}{4 \delta_s B^2 p^2 (x_2 - x_1) \left[ \frac{\pi}{2B} (y_2 - y_1) - \frac{1}{4} \sin\left(\frac{2\pi}{B} y_2\right) + \frac{1}{4} \sin\left(\frac{2\pi}{B} y_1\right) \right]} \quad (28)$$

B. If  $(x_2 - x_1) \cdot (y_2 - y_1) = \delta \ll (A \cdot B)$  and  $(y_1 + y_2)/2 = y_0$ .

$$Q_s = AC [B^2 p^2 + C^2] / \delta_s (4Bp^2) \sin^2\left(\frac{\pi}{B} y_0\right) \cdot \delta \quad (29)$$

Thus we can write  $\delta_s$  as a function of  $Q_s$ , the cavity geometry, the sample position, and the normal mode, all of which can be found from experimental values. Equations 28 or 29 were used to calculate the values of  $\delta_s$  presented in Tables 2 and 3 of this report.

Finally, the uncertainty in  $\delta_s$  is given for large samples by

$$\Delta\delta_s = \frac{\delta_s}{Q_s} \Delta Q_s + \left[ \frac{\pi AC [p^2 B^2 + c^2]}{Q_s 4B^2 p^2 (x_2 - x_1) \left[ \frac{\pi}{2B} (y_2 - y_1) - \frac{1}{4} \sin \left[ \frac{2\pi}{B} (y_2 + \Delta y) \right] + \frac{1}{4} \sin \left[ \frac{2\pi}{B} (y_1 + \Delta y) \right] \right]} - \delta_s \right] \quad (30)$$

and for small samples by

$$\Delta\delta_s = \frac{\delta_s}{Q_s} \Delta Q_s + \left[ \frac{AC [B^2 p^2 + c^2]}{Q_s 4B^2 p^2 \cdot S \cdot \sin^2 \left[ \frac{\pi}{B} (y_0 + \Delta y) \right]} - \delta_s \right] \quad (31)$$

where  $\Delta y$  is the uncertainty of the position of the sample along the  $y$  axis in the cavity.

Equation (31) can be used to estimate the dependence of the sensitivity of substitution techniques on flake size and skin depth. We calculate the values of the second term (in the brackets) to pass through the 10% value for combinations such as:

$$\delta = 25.0 \text{ microns with } \Delta x \sim \Delta y \sim 0.5 \text{ mm}$$

$$\text{and } \delta = 2.5 \text{ microns with } \Delta x \sim \Delta y \sim 2 \text{ mm}$$

$$\text{and } \delta = 0.25 \text{ microns with } \Delta x \sim \Delta y \sim 5 \text{ mm}$$

This uncertainty (by Eq. 31) rises steeply for smaller semiconductor flakes and is only a few percent, for larger ones.

## APPENDIX C

## CAVITY LOADING BY SEMICONDUCTOR END WALL

- X = The reflective cavity with choke and the sample as an end wall
- Y = The reflective cavity with choke and a flat piece of Cu as an end wall
- Z = The reflective cavity with equal dimensions to X and Y but without a choke
- T = The transmission cavity
- $Q_{c,1}$  = The Q of the first coupling iris
- $Q_{c,2}$  = The Q of the second coupling iris
- $Q_{R,CH}$  = The Q of the choke joint due to microwave energy being radiated out of the slots of the choke
- $Q_{w,s}$  = The Q of the sample end wall
- $Q_{w,X}$  = The Q of the X cavity due to ohmic losses in the walls of the cavity except for the sample which acts as an end wall
- $Q_{w,Y}$  = The Q of the Y cavity due to ohmic losses in the walls of the cavity except for the Cu flat, which acts as an end wall
- $Q_{w,Z}$  = The Q of the Cu flat acting as an end wall in the Y cavity
- $Q_{w, end}$  = The Q of the end wall of the Z cavity
- $\beta_{1,\underline{\mu}}$  = The coupling parameter of the first coupling iris as measured in the  $\underline{\mu}$  cavity
- $\beta_{2,\underline{\mu}}$  = The coupling parameter of the second coupling iris as measured in the  $\underline{\mu}$  cavity

## H. Equations for cavity X

$$1B. \frac{1}{Q_{c,1}} + \frac{1}{Q_{R,CH}} + \frac{1}{Q_{w,s}} + \frac{1}{Q_{w,X}} = \frac{1}{Q_{L,X}}$$

$$2B. \frac{1}{Q_{R,CH}} + \frac{1}{Q_{w,s}} + \frac{1}{Q_{w,X}} = \frac{1}{Q_{u,X}}$$

$$3B. \beta_{1,X} Q_{c,1} = Q_{u,X}$$

## I. Equations for cavity Y

$$4B. \frac{1}{Q_{c,1}} + \frac{1}{Q_{R,CH}} + \frac{1}{Q_{w,Cu}} + \frac{1}{Q_{w,Y}} = \frac{1}{Q_{L,Y}}$$

$$5B. \frac{1}{Q_{R,CH}} + \frac{1}{Q_{w,Cu}} + \frac{1}{Q_{w,Y}} = \frac{1}{Q_{u,Y}}$$

$$6B. \beta_{1,X} Q_{c,1} = Q_{u,Y}$$

## J. Equations for cavity Z

$$7B. \frac{1}{Q_{c,1}} + \frac{1}{Q_{w,Z}} = \frac{1}{Q_{w,Z}}$$

$$8B. \beta_{1,Z} Q_{c,1} = Q_{w,Z}$$

## K. Equations for cavity T

$$9B. \frac{1}{Q_{c,1}} + \frac{1}{Q_{w,T}} + \frac{1}{Q_{c,2}} = \frac{1}{Q_{L,T}}$$

$$10B. Q_{L,T} = \frac{f_o}{\Delta f(3db)}$$

$$11B. Q_{w,T} = Q_{L,T}(\beta_{1,T} + \beta_{2,T} + 1)$$

In a rectangular mode  $Q_{R,CH}$  is small enough that it cannot be ignored in the above equations. Therefore, a final solution to  $Q_{w,s}$  must take into account the value of  $Q_{R,CH}$

If we assume that  $Q_{w,z}$  is equal to  $Q_{w,T}$ , then, using equations 8B. and 11B. we get

$$12B. \quad Q_{c,1} = \frac{Q_{L,T}(\beta_{1,T} + \beta_{2,T} + 1)}{\beta_{1,Z}} \quad \text{and thus,}$$

$$13B. \quad Q_{u,X} = \frac{(\beta_{1,X})Q_{L,T}(\beta_{1,T} + \beta_{2,T} + 1)}{(\beta_{1,Z})}$$

also, since

$$\frac{1}{Q_{u,X}} = \frac{1}{Q_{R,CH}} + \frac{1}{Q_{w,s}} + \frac{1}{Q_{w,Y}}$$

and

$$\frac{1}{Q_{w,end}} + \frac{1}{Q_{w,X}} = \frac{1}{Q_{w,z}} \quad \text{thus,}$$

$$14B. \quad \frac{1}{Q_{u,X}} = \frac{1}{Q_{R,CH}} + \frac{1}{Q_{w,s}} + \frac{1}{Q_{w,Z}} - \frac{1}{Q_{w,end}}$$

As  $(K)Q_{w,Z}$  is equal to  $Q_{w,end}$ , where  $K$  is a constant determined by the geometry of the cavity, we get

$$15B. \quad \frac{1}{Q_{u,X}} = \frac{1}{Q_{R,CH}} + \frac{1}{Q_{w,s}} - \frac{1}{(K)Q_{w,Z}} + \frac{1}{Q_{w,Z}}$$

To solve for  $\frac{1}{Q_{R,CH}}$ , we assume that the losses in the Cu end wall piece of cavity Y are equal to the losses in the end wall of cavity Z, so that  $Q_{w,Cu}$  is equal to  $Q_{w,end}$ , then

$$16B. \quad \frac{1}{Q_{w,Z}} = \frac{1}{Q_{w,Cu}} + \frac{1}{Q_{w,X}} \quad \text{using equations 5B, 6B, 12B,}$$

and 16B., we get

$$17B. \quad \frac{1}{Q_{R,CH}} = \frac{(\beta_{1,Z})}{Q_{L,T}(\beta_{1,T} + \beta_{2,T} + 1)(\beta_{1,X})} - \frac{1}{Q_{w,Z}}.$$

Substituting 17B. into 15B. and using equations 11B. and 13B yields

$$18B. \quad \frac{1}{Q_{w,s}} = \frac{(\beta_{1,Z})}{Q_{L,T}(\beta_{1,T} + \beta_{2,T} + 1)} \left[ \frac{1}{\beta_{1,X}} - \frac{1}{\beta_{1,X}} + \frac{1}{(K)\beta_{1,Z}} \right]$$

$$\text{Now } K = \frac{Q_{w,end}}{Q_{w,Z}} = \frac{\int_{sw} H_T^2 ds}{\int_{se} H_T^2 ds} \quad \text{where } sw$$

indicates that the integral is taken over all the surface area of the cavity, se indicates that the integral is taken over the end wall only, and  $H_T$  is the magnitude of the tangential magnetic field. In fact,

$$K = \frac{[2C^3A + c^3B + p^2B^3C + 2Ap^2B^3]}{AB^3p^2} \quad \text{where } A, B, \text{ and } C \text{ are}$$

the dimensions of the Z cavity, and  $p = \frac{2C}{\lambda g}$

$$\text{Finally, as } Q_{w,s} = \frac{2 \int_v H^2 dv}{\delta_s \int_{se} H_T^2 ds} \quad \text{we get}$$



$$19B. \quad \delta_s = \frac{[B^2 p^2 + C^2] C}{Q_{w,s} B^2 p^2} \quad \text{and}$$

$$\Delta \delta_s = \frac{[B^2 p^2 + C^2] C}{B^2 p^2 (Q_{w,s})^2} \Delta(Q_{w,s}) = \frac{\delta_s}{Q_{w,s}} \cdot \Delta(Q_{w,s}).$$

## APPENDIX D

## GaAs PL DATA REDUCTION

(by J. M. Rowe)

A computer program has been used to aid in correcting photoluminescence data for the response of the optical system, photomultiplier tube and associated electronics. Calibration factors obtained by running a tungsten ribbon lamp and using the known emissivity of tungsten had been tabulated by wavelength every 5 Å between 8010 Å and 8920 Å and were read in by the computer. The remainder of the input consisted of chart reference marks and data points in chart coordinates, all taken from the record produced during a data run. Since the computer used data in chart paper coordinates, much work was saved.

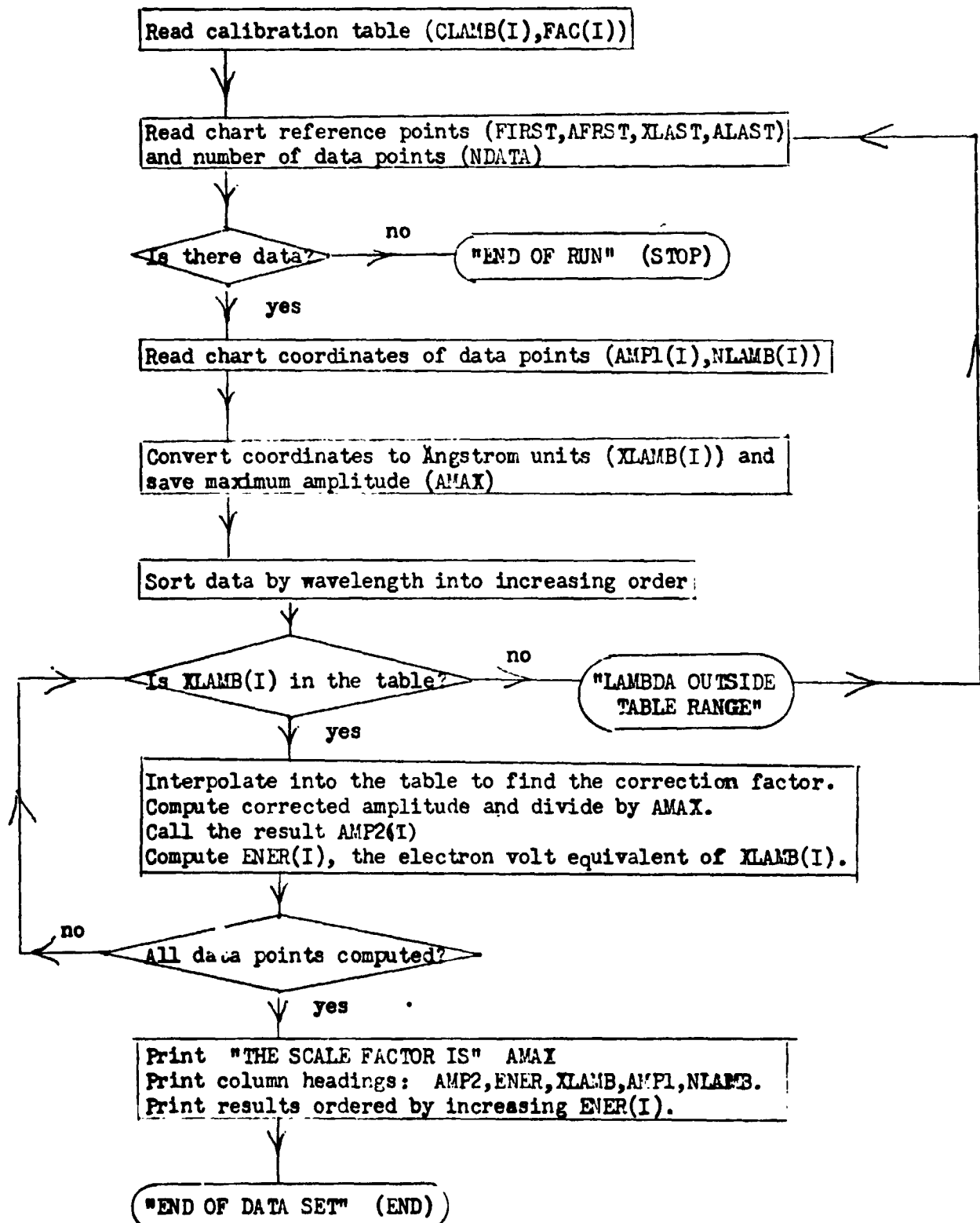
During data taking, the wavelength in Ångstroms was noted at the beginning and end of a run and these points were marked on the chart paper. These wavelengths and their corresponding coordinates were read into the computer and using this the computer determined the wavelengths in Ångstroms of data points. This coordinate was taken as an integer for convenience. PL amplitude coordinates were taken as floating point numbers and were arbitrary to within a scale factor written down during data taking in case it would be needed later.

Each data wavelength was found in the table using linear interpolation and the interpolated correction factor applied. Corrected amplitudes were each divided by the maximum uncorrected amplitude for the data set to yield comparable scales. This was done for convenience. The scale factor appears in the printout and no information was lost. The electron volt equivalent of each wavelength was also computed.

The output appears in tabulated form with five columns. These are:

- (1) corrected and scaled amplitude, (2) energy in electron volts, (3) wavelength in  $\text{\AA}$ , (4) uncorrected amplitude, and (5) chart coordinate of wavelength. The listing was made in order of increasing energy, or alternatively fed to Karla Dalton's program for display and for automatic graphing at the Tektronix terminal of the UNIVAC 1108 computer.

The flow chart for the PL DATA REDUCTION program is given on the next page. A listing of the program and one output table is attached, too.

FL Data Reduction Flow Chart

GLOSSARY (By order of appearance)

CLAMB(I)	wavelength in the calibration table
FAC(I)	calibration factor at wavelength CLAMB(I)
FIRST	wavelength ( $\text{\AA}$ ) of initial chart reference mark
AFRST	chart coordinate of initial chart reference mark
XLAST	wavelength ( $\text{\AA}$ ) of final chart reference mark
ALAST	chart coordinate of final chart reference mark
NDATA	number of data points for the chart
AMP1(I)	chart coordinate of the PL amplitude
NLAMB(I)	chart coordinate of wavelength (integer) corresponding to AMP1(I)
XLAMB(I)	wavelength ( $\text{\AA}$ ) corresponding to NLAMB(I)
AMAX	maximum uncorrected amplitude found in the data set
AMP2(I)	corrected amplitude divided by AMAX
ENER(I)	energy (eV) corresponding to XLAMB(I)

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,ST
U 02/27-10123125-(, )
000 BASG,T 10,T
000 BSAVT 10,KARLADALTON CASTLE PL DATA REDUCTION
000 B FOR,X1ST GAAS
000 C G(A)A(S) PL DATA REDUCTION
000 DIMENSION CLAMB(500),FAC(500),AMP1(300),NLAMB(300)
000 DIMENSION XLAMB(300),AMP2(300),ENER(300)
000 10 FORMAT (15)
000 20 FORMAT (8F10.2)
000 25 FORMAT (4F10.2,15)
000 30 FORMAT (F10.2,15,F10.2,215)
000 40 FORMAT (F10.2,15)
000 READ 10,ITBL
000 READ 20,(CLAMB(J),FAC(J),CLAMB(J+1),FAC(J+1),CLAMB(J+2),FAC(J+2),
000 CLAMB(J+3),FAC(J+3), J=1,ITBL,4)
000 1 READ 25,FIRST,AFRST,XLAST,ALAST,NDATA
000 IF (NDATA.GT.0) GO TO 3
000 PRINT 90
000 90 FORMAT (1X,1)HEND OF RUN.)
000 STOP
000 3 READ 40,(AMP1(J),NLAMB(J),J=1,NDATA)
000 DIFN = ALAST - AFRST
000 UNIT = (XLAST - FIRST)/DIFN
000 AMAX = AMP1(1)
000 DO 100 I = 1,NDATA
000 XLAMB(I) = (NLAMB(I) - AFRST)*UNIT + FIRST
000 100 IF (AMP1(I).GT.AMAX) AMAX = AMP1(I)
000 NDM1 = NDATA - 1
000 DO 400 I = 1,NDM1
000 IP1 = I + 1
000 DO 400 J = IP1,NDATA
000 IF (XLAMB(I).LE.XLAMB(J)) GO TO 400
000 TEMP = XLAMB(I)
000 XLAMB(I) = XLAMB(J)
000 XLAMB(J) = TEMP
000 TEMP = NLAMB(I)
000 NLAMB(I) = NLAMB(J)
000 NLAMB(J) = TEMP
000 TEMP = AMP1(I)
000 AMP1(I) = AMP1(J)
000 AMP1(J) = TEMP
000 400 CONTINUE
000 N = 1
000 DO 200 I = 1,NDATA
000 DO 300 J = N,ITBL
000 IF (XLAMB(I).GE.CLAMB(J)) GO TO 300
000 N = J
000 C=(XLAMB(I)-CLAMB(J-1))/(CLAMB(J)-CLAMB(J-1))
000 C=C*(FAC(J)-FAC(J-1))/FAC(J-1)
000 AMP2(I)=AMP1(I)+C/AMAX
000 ENER(I) = 1.2395E4/XLAMB(I)
000 GO TO 200
000 300 CONTINUE
000 PRINT 50
000 50 FORMAT (1X,27)HLAMBDA OUTSIDE TABLE RANGE.)
000 GO TO 1

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000 200 CONTINUE
000 PRINT 55, AMAX
000 55 FORMAT(1X, ' THE SCALE FACTOR IS ', F10.4)
000 2 PRINT 60
000 WRITE(10)NDATA, (ENER(I), AMP2(I), XLAMB(I), AMPI(I), I=1, NDATA)
000 PRINT 70 (AMP2(I), ENER(I), XLAMB(I), AMPI(I), NLAMB(I),
000 I=1, NDATA), -17
000 PRINT 80
000 60 FORMAT(17X, 4HAMP2, 6X, 4HENER, 5X, 5HXLAMB, 6X, 4HAMPI, 2X, 5HNLAMB/)
000 70 FORMAT(11X, 2F10.4, 2F10.2, 15I)
000 80 FORMAT(1X, 16HEND OF DATA SET.//)
000 END
000 BXQT
000 184
000 8010. 1.000 8015. 1.006 8020. 1.013 8025. 1.021
000 8030. 1.028 8035. 1.037 8040. 1.045 8045. 1.052
000 8050. 1.060 8055. 1.067 8060. 1.073 8065. 1.081
000 8070. 1.089 8075. 1.098 8080. 1.106 8085. 1.115
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000 8270. 1.397 8275. 1.404 8280. 1.411 8285. 1.420
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000 8470. 1.779 8475. 1.793 8480. 1.807 8485. 1.821
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000	15.7	58
000	15.0	59
000	14.5	60
000	13.9	61
000	13.2	62
000	12.7	63
000	12.1	64
000	11.7	65
000	11.0	66
000	10.7	67
000	10.3	68
000	9.8	69
000	9.6	70
000	9.3	71
000	8.9	72
000	8.6	73
000	8.4	74
000	8.0	75
000		

HOWE - ACCT UAHXXXXXXXXXX- PROJECT: - QSQSQS-